IS 3562 (1997): p-Nitrotoluene, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS 3562 : 1997
(Reaffirmed 2002)

भारतीय मानक

पी-नाइट्रोटोल्यून, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

p-NITROTOLUENE, TECHNICAL — SPECIFICATION

(First Revision)

ICS 71.080.30

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1997

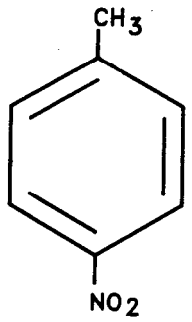
Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1965. Subsequently, the Committee responsible for preparation of this standard felt the need to revise it to keep it in line with present industrial practices. In this revision, all the requirements have been modified, the method for assay of *p*-nitrotoluene has been changed to gas chromatography

p-Nitrotoluene ($C_7H_7NO_2$) or 4-Nitrotoluene or 1-Methyl-4-Nitrobenzene is an intermediate used for the manufacture of *p*-toluidine, fuchsine and various other synthetic dyes, apart from its use in the manufacture of dinitrostilbene compounds. It is also a drug intermediate being the starting material for the preparation of *p*-nitrobenzoic acid which is used in the preparation of synthetic drugs like folic acid. It has the following structural formula:



p-NITROTOLUENE
(Molecular Mass - 137)
CAS Registry No. [99-99-0]

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
p-NITROTOLUENE, TECHNICAL — SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for *p*-nitrotoluene, technical.

2 NORMATIVE REFERENCE

The following Indian Standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties of agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (*first revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests of dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of yellowish solid with unpleasant odour and free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

Unless otherwise agreed to, the material shall be packed in steel drums (*see* IS 2552) lined with a suitable plastic film.

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Indication of the source of manufacture;

c) Lot or batch number;

d) Net mass; and

e) POISON [In accordance with IS 1260 (Part 1)]

Table 1 Requirements for *p*-Nitrotoluene

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C *Min*	51.0	7 of IS 5299
ii)	Assay, by GC, percent by mass, *Min*	99.0	Annex A
iii)	Dinitrocresols	To pass the test	Annex B
iv)	*o*-Nitrotoluene isomer, percent by mass, *Max*	0.1	Annex A
v)	*m*-nitrotoluene isomer, percent by mass, *Max*	0.5	Annex A
vi)	Dinitrotoluene, percent by mass, *Max*	0.1	Annex A
vii)	Unnitrated hydrocarbons, percent by mass, *Max*	0.1	Annex A
viii)	Matter insoluble in methanol, percent by mass, *Max*	0.1	**10.3** of IS 5299

4.2.1 Each container shall, in addition, bear the minimum cautionary notice worded as under:

'AVOID CONTACT WITH SKIN;
DO NOT HEAT ON A NAKED FLAME'

4.2.2 *BIS Certification Marking*

Each container may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in 3 of IS 5299.

5.2 Number of Tests

5.2.1 Tests for description, crystallizing point, assay and impurities, that is, *o*-nitrotoluene isomer, *m*-nitrotoluene isomer, dinitrotoluene and unnitrated hydrocarbons shall be done on each sample.

5.2.2 Test for the determination of dinitrocresols and matter insoluble in methanol shall be performed on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of the description, crystallizing point, assay and impurities, that is, *o*-Nitrotoluene isomer, *m*-nitrotoluene isomer, dinitrotoluene and unnitrated hydrocarbons, if each of the individual test results satisfies the relevant requirements given in **3.1** and Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirement of dinitrocresols and matter insoluble in methanol tested on the composite sample the test result shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (ii), (iv), (v), (vi) *and* (vii)]

GAS CHROMATOGRAPHIC ANALYSIS FOR *p*-NITROTOLUENE

A-1 GENERAL

The contents of *p*-nitrotoluene, *o*-nitrotoluene, *m*-nitrotoluene, dinitrotoluenes and unnitrated compounds are determined by gas chromatographic analysis.

A-1.1 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, acetone) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the detector end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column. The detector signals, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-2 APPARATUS

A-2.1 Any suitable gas chromatograph and column capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The conditions for a typical chromatographic analysis of *p*-nitrotoluene are given below:

Sample	*p*-Nitrotoluene (10 percent solution in acetone)

Injection quantity	0.5 microlitres
Column	
Material	Glass
Length	4 m
Orifice	0.63 cm OD, 0.2 cm ID
Packing material	OV 17 + QF 1 (1.5% + 1.5%)on Chromosorb WHP, 80-100 Mesh
Carrier Gas	Nitrogen
Conditions	
Carrier flow rate	30 ml/min
Oven temperature	110°C -18 minutes hold
	– 10°C/min programming to –180°C hold 20 minutes
Injection port temperature	250°C
Detector	
Type	F.I.D.
Temperature	250°C
Data start time	1.5 minutes
Data end time	40 minutes

A-2.2 Interpretation of Chromatogram

Elution order of components is as follows:

a) Acetone (solvent used),

b) Toluene,

c) Nitrobenzene,

d) *o*-Nitrotoluene,

e) *m*-nitrotoluene,

f) *p*-nitrotoluene,

g) 2,6 Dinitrotoluene, and

h) 2,4 Dinitrotoluene.

A-3 CALCULATION

A-3.1 Calculate the concentration of individual constituents from the areas of specific constituents shown on the chromatogram of the material on the basis of peak areas on chromatogram obtained with known amount of pure constituents using the same apparatus under identical condition.

A-3.2 Area Measurement (*See* **Note**)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height times the width of half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-3.3 Area Normalization

By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE — Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator if fixed with GC machine, would be of great advantage.

ANNEX B

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF DINITROCRESOLS

B-1 PRINCIPLE

Dinitrocresols being soluble in sodium carbonate solution impart characteristic colour to the solution. This colour is compared with that obtained with a known quantity of dinitrocresol.

B-2 REAGENTS

B-2.1 Sodium Carbonate Solution — 10 percent (*m/v*), approximately.

B-2.2 Standard *o*-Nitro *p*-Cresol Solution

Dissolve 50 mg of vaccum distilled *o*-nitro *p*-cresol in 1 000 ml of 10 percent sodium carbonate solution.

B-3 PROCEDURE

Wash 100 g of the material with water till the last washing is neutral to litmus. Shake the washed material very quickly with 100 ml of sodium carbonate solution for one minute. Separate the alkaline solution from the material and repeat the above process of shaking with sodium carbonate solution. Compare the colour thus obtained with that obtained with the standard solution of *o*-nitro *p*-cresol.

The material shall deemed to have passed the test if the colour obtained is not deeper than the standard *o*-nitro *p*-cresol solution.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 11 (1105).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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